

# BIOMATERIALS

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## PHYSICAL-MECHANICAL PROPERTIES OF COMPOSITE LIGHT-CURED MATERIALS

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Composite stomatological light-cured materials with a polymer matrix, bonding layer and inorganic filling (glass) are examined. The requirements imposed on these materials by modern therapeutical stomatology are indicated. The composition and some technological aspects of the production of the new material are presented. An investigation of the physical-mechanical properties of the new composite material (strength, water absorption, polymerization depth) showed that these indices are on par with the best foreign analogs.

**Key words:** composite material, glass (filler), polymer matrix, strength, water absorption, polymerization.

Light-cured stomatological materials comprised of a polymer matrix, inorganic filler (glass) and an adhesive, which plays the role of a bonding layer, are in greatest demand at present, but in the Republic of Belarus they are not produced and the demand for them is filled by imports. The cost of the imported analogs, which is  $7.5 \times 10^3$  US\$/kg on average, is holding back wide adoption of such materials in stomatological practice.

There is virtually no information about the compositions and technologies used to obtain the light-cured imported analogs. The available data on the materials composition and structure of the polymer component are also sporadic.

Our objectives in the present work are to develop and investigate the physical-mechanical properties of a composite light-cured material.

Composite materials must possess the following characteristics: high mechanical strength; formation of a chemical bond with dental tissue (enamel, dentine); gluing of materials by fragments (composite – composite, composite – compomer, composite – glass-ionomer material, and so on); biological tolerance of materials (third-, fourth- and fifth-generation adhesive systems, high degree of polymerization); similitude to natural dental tissues (owing to physical properties: strength, thermometric expansion, color, opaqueness, wear

resistance, water absorption); stability and insolubility in oral fluids; and, possibility of restoring teeth containing defects of different forms and origin.

General use of composites became possible after R. L. Bowen introduced into practice bisphenol glycidyl methacrylate (BIS-GMA). This monomer possesses a high molecular mass and it is capable of forming very long chains, which ‘encompass’ small particles of filler. BIS-GMA is the basis of almost all modern stomatological composites. Polymerization initiators and inhibitors, catalysts, UV radiation absorbers and other substances are also present in the organic matrix [1].

Research on the modification of composites is focused on reducing the consequences of shrinkage, improving biocompatibility, durability and practicability [2]. A polymer organic matrix usually includes acrylate or methacrylate monomers containing at least one unsaturated double bond and can also contain oligomers and polymers. Curing of these composite materials is initiated by visible blue light with wavelength 450 – 550 nm.

The polymer matrix determines the plasticity, adhesive properties and biocompatibility of the composite and it affects the strength, color stability and degree of polymerization. The magnitude of the shrinkage and other characteristics depend on the volume of the organic material.

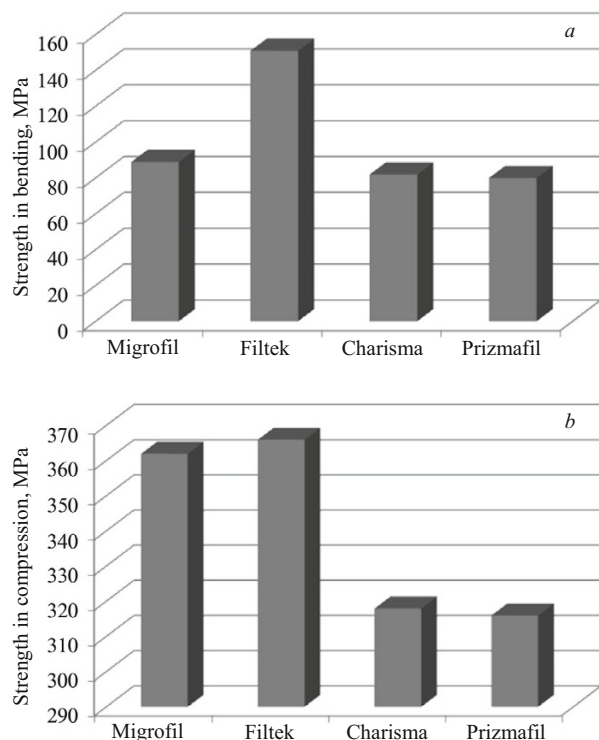
A novel composition of the polymer matrix, introduced in the amount 20%<sup>4</sup> into the composite, was developed to-

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<sup>4</sup> Here and below, unless stipulated otherwise, the content by weight, %.



**Fig. 1.** Strength of samples of composite materials in bending (*a*) and compression (*b*).

gether with the Grodno Scientific-Research and Design Institute of the Nitrogen Industry and Products of Organic Synthesis. After the components of the matrix are mixed the mixture is loaded into a UV irradiation chamber for further polymerization.

The following properties depend on the inorganic filler (whose content in the composite reaches 80%): strength, shrinkage, wear resistance, x-ray contrast and color stability. Glass powder with a definite composition is used as the inorganic filler. The glasses most often encountered are  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{BaO}$ ,  $\text{B}_2\text{O}_3$ ,  $\text{SrO}$ ,  $\text{ZrO}_2$  and others.

Systematic research on obtaining the optimal composition of glass in the three-component system  $\text{SiO}_2$ – $\text{Al}_2\text{O}_3$ – $\text{B}_2\text{O}_3$ , additionally modified by F, SrO and  $\text{WO}_3$ , was performed during the development of the composition. For this the components of the batch were prepared by mixing and comminuting and glass was synthesized in an electric furnace in a definite regime followed by air cooling of the melts. The cooled glass was subjected to grinding in a planetary mill with uralite grinding bodies and the comminuted material was passed through a sieve. Depending on the grinding regime fillers with different granulometric composition were obtained. On the whole the filler particle size can vary from 0.01 to 45  $\mu\text{m}$ . The larger the particles, the more filler can be added into the composition of the composite and the greater the strength of the material and the lower the shrinkage are. However, particles which are too large form a rough, lusterless surface and give a composite material with

high abrasion resistance. Small particles make it possible to obtain a polishable composite which is more durable, but as a result of the larger specific surface area only a limited number of particles can be introduced into the composite. In materials with small filler particles the main physical indices of the composite, such as strength, water absorption and color stability, are degraded.

The following composition of an inorganic filler (80%) was developed in [3, 4]: glass filler ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ , F, SrO,  $\text{WO}_3$ ), treated with silane in order to improve the strength characteristics of the restorative material and hydrophobic aerosol DT4. Strontium oxide used as an x-ray contrast material makes the glass more stable. Such filler gives the composite material high aesthetic properties. The CLTE of the new filler (glass-containing material) best matches the CLTE of the polymer matrix.

The components of the filler particle and polymer matrix were mixed in a planetary mill, where the mix obtained was subjected to evacuation in order to remove the air bubbles at temperature no higher than 40°C.

The physical-mechanical properties of the new composite material ‘Migrofil’ (strength in compression and bending, water absorption, depth of polymerization) were determined under identical test conditions following GOST R 51202–98 requirements [5] relative to the best imported analogs Filtek Z250 3M ESPE (USA), Charisma Heraeus/Kulzer (FRG) and ‘Prizmafil’ (StomaDent JSC, RF) (Figs. 1 and 2).

Mechanical strength is the most important characteristic of a composite material, since it imparts shape stability and abrasion resistance. It was determined by subjecting a 6 mm in diameter, 3 mm thick shortened cylinder to uniform compression to total fracture with the load bar of an Instron machine moving at the rate 10 mm/min.

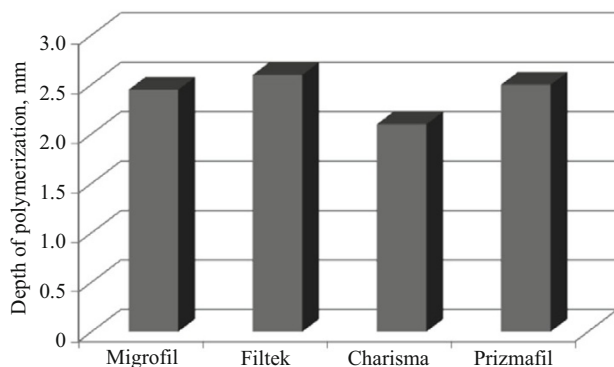
As one can see from Fig. 1*a*, the strength in bending of the Migrofil material is 90 MPa, which meets the standard requirements [5] and is higher than that of the Charisma and Prizmafil analogs, but 60 MPa lower than for the composite Filtek. The strength in compression (Fig. 1*b*) of Migrofil is practically the same as that of the American analog, 360 and 365 MPa, respectively, while the Russian and German analogs fall short by 45 MPa.

The volume expansion of the light-cured composite materials is an important property, since on the one hand it lowers the polymerization stress, but on the other hand large volume expansion can result in excess restorative stress. The volume expansion in water (or water absorption) of most light-cured composite materials is about 1%.

The material samples prepared in the form of 15 mm in diameter, 0.5 mm thick disks were immersed in distilled water at temperature 37°C for 7 days.

The water absorption of all test samples was 0.98  $\mu\text{g}/\text{mm}^3$ . This corresponds to the standards [5].

The composite light-cured stomatological materials exhibit different polymerization shrinkage and stress. However, it should be noted that composites with a large polymeriza-



**Fig. 2.** Degree of polymerization of the samples of composite materials.

tion depth make it impossible to introduce a large quantity of material during restorative work because the composite can detach from the wall of the tooth cavity. The use of such composites can result in different kinds of complications (chips, cracks, color change and so on).

To determine the polymerization depth the experimental material was placed in a 6 mm long, 5 mm in diameter metal mold and polymerized by using a lamp with light flux intensity  $800 \text{ mW/cm}^2$  for 20 sec. Then the samples were extracted from the mold and the unpolymerized part of the material was removed. The height of the solidified cylindrical sample was measured with a micrometer to within  $\pm 0.1 \text{ mm}$ .

As one can see from Fig. 2, the composite Charisma exhibits the smallest polymerization depth (2.1 mm), while the

other test samples have practically the same indices, which also corresponds to the specifications in [5].

In summary, the test results obtained for the physical-mechanical properties of the new light-cured composite material Migrofil, in which the content of the glass-based filler is 80%, showed correspondence to the foreign analogs and the regulatory requirements.

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